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Biodiesel production from renewable feedstocks: Status and opportunities

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ABSTRACT

The increased demand for energy, climate change, and energy security concerns has driven the research interest for the development of alternative fuel from plant origin. Biodiesel derived from plant oils, which include edible and non-edible oil have gained interest for the last two decades as alternative for diesel around the world. Among these plant origin oils more than 95% of biodiesel production feedstocks come from edible oils, because they are readily available in many regions. The major advantage of these feedstocks is the properties of biodiesel produced from them are suitable to be used as diesel fuel substitute. But the consequence is the increase demand of the feedstock for food as well as fuel. A sustainable alternative fuel should be derived from renewable non-food biomass sources. The main objective of this review is to give an overview on the synthesis of biodiesel through esterification and transesterification using non-edible oil resources which are available in India, and available processes for synthesis of biodiesel (acid-, base-catalyzed transesterification reactions (homogeneous and heterogeneous), their importance, and which is the commercial process also discussed here.

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1. Introduction

Fuels are playing a major role in the economy of every country of this world and majority of the world energy needs are supplied by the petrochemical resources, coal and natural gases, with the exception of hydroelectricity and nuclear energy [1,2]. Among these mineral oils, petroleum plays a major role in the development of industrial growth, transportation, agricultural sector and to meet many other basic needs of human being [3]. Since 1970s mineral-based fuel oil (i.e. petroleum, diesel, kerosene, natural gas, etc.) prices are increasing day by day due to depletion of fossil fuel reserves and rapid consumption of mineral oils. The main reason that caused the fast diminishing of energy resources is due to rapid population and industrialization growth globally [4]. Moreover petroleum-based fuels are creating negative impact on the ecosystem and burning of these fuels leads to emission of pollutant gases like CO_2, HC, NOx, SOx [5]. Due to the above and following reasons like rapidly increasing prices, uncertainties concerning petroleum availability, increased environmental concern and effect of green house gases from industries have stimulated the search for alternative sources for petroleum-based fuel including diesel fuel [6].

As a result, biodiesel and ethanol has been known as bio-fuel to substitute petroleum derived cetane and octane fuel, respectively. These fuels have been attracted the attention, since use of these oxygenated fuels in the engines clearly reducing the exhaust emission of green house gases, particulate matters, unburned hydrocarbons, poly-aromatics and oxides of sulfur [7].

Biodiesel a processed fuel derived from the vegetable oils and animal fats through the esterification and transesterification reactions of free fatty acids (FFAs) and triglycerides, respectively, that occur naturally in renewable biological sources [8]. In other words we can define biodiesel is mixture of alkyl esters of long chain fatty acids, which are synthesized through esterification and transesterification of free fatty acids (FFAs) and triglycerides (TG) [2,8,9]. The major feedstocks available for biodiesel are rapeseed, palm, canola and soybean oils, though the process to grow non-food grade oil is under process in the developing nations in tropic and sub-tropics. Many bio-diesel industries have been set in the last decades worldwide, whereas most of these are not operational throughout the year due to the scarcity of cheap vegetable oils as feedstock for economic production of biodiesel.

In the production of biodiesel more than 95% of feed stocks come from edible oils since they are mainly produced in many regions of the world and the properties of biodiesel produced from these oils are much suitable to be used as diesel fuel substitute. Use of such edible oil to produce biodiesel is not feasible in view of a big gap in demand and supply of such oils as food and they are far expensive to be used at present and obviously, the use of non-edible vegetable oils compared to edible oils is very significant. Moreover biodiesel does not contain any compounds like sulfur or aromatic compounds and burning of biodiesel results in lower emission of hydrocarbons, carbon monoxides and particulate matters [10,11]. Since the cost of raw materials accounts about 60–80% of the total cost of biodiesel production, so choosing a right feed stock is very important and properties of biodiesel produced from different feed stocks would be quite different [2]. However, it may cause some problems such as the competition with the edible oil market, which increases both the cost of edible oils and biodiesel; moreover it will cause deforestation in some countries because more and more forests have been felled for plantation purposes. In order to overcome these disadvantages, many researchers, scientists, technologists as well as industrialists are interested in non-edible oil source which are not suitable for human consumption because of the presence of some toxic components in the oils. Furthermore, non-edible oil crops can be grown in wastelands that are not suitable for food crops and the cost of cultivation is much lower because these crops can still sustain reasonably high yield without intensive care [2,6,12]. However, most non-edible oils contain high free fatty acids. Thus they may require multiple chemical steps or alternate approaches to produce biodiesel, which will increase the production cost, and may lower the ester yield of biodiesel below the standards.

In recent days a number of methods are available and have been adopted for the production of biodiesel. There are four primary ways to produce biodiesel such as direct use and blending of raw oils, micro-emulsions, thermal cracking and transesterification [2]. The purpose of transesterification process is to lower the viscosity of the oil. The transesterification is the process of removing the glycerides and combining oil esters of vegetable oil with alcohol. The process reduces the viscosity to a value comparable to that of diesel and hence improves combustion [3]. This chemical process converts the triglycerides into fatty acid methyl esters (FAMEs), by releasing glycerol as a by-product. Currently the main synthetic approaches used for biodiesel production includes base-catalyzed transesterification, acid-catalyzed transesterification (with simultaneous esterification of free fatty acids), non-catalytic conversion via transesterification and esterification under super critical alcohol conditions, microwave and ultrasound [8]. Recently heterogeneous base-catalyzed transesterification gaining more interest due to its great advantages towards product separation, reusability of catalyst and reaction conditions. Above all each catalytic and non-catalytic process has its own importance and advantages towards esterification and transesterification reactions. Furthermore, in both acid- and base-catalyzed approaches, conversion efficiency is highly dependent upon the water and free fatty acid content of the feedstocks. Therefore, in this paper attempt has been made to give an overview on the synthesis of biodiesel through esterification and transesterification using non-edible resources which are available in India, and available processes for synthesis of biodiesel (homogeneous and heterogeneous), their importance, and which is the commercial process also we discussed here [4,13–19].

2. Feedstock's for biodiesel

Since last few years many biodiesel production plants have been identified in India. These plants are operational as per the availability of feedstock, price of crude vegetable oils. The common feed stock for the production of biodiesel in India is palm oil, which is being imported from Malaysia and Indonesia. As palm oil is being used for edible purpose, the price of the crude palm oil fluctuates in the international market. Apart from that some other feedstocks are also being used (i.e. fish oil, used cooking oil) but to a very less extent. Even though the consumption of edible oils in some countries like India is high, the availability of used cooking oil is very small as it is used till the end. Hence, focus needs to be

shifted to non-edible oilseed plants available in India and the details of such potential oilseed plant are shown in Table 1. Among these non-traditional oilseeds plants huge scope exists for the oilseeds such as karanja and jatropha as biodiesel feedstock. Since these two plants grow in adverse agro-climatic conditions, less gestation period (about 2–3 years for jatropha and 5 years for karanja) and their fatty acid composition suitable for producing quality biodiesel. The oil from algal biomass has also attracted the attention as the future feedstock for biodiesel. In this review an attempt has been made to provide an information on the different non-edible/waste oil resources which is available in India namely castor oil, cottonseed oil, jatropha, jojoba, karanja, kokum, mahua, nahor, neem, palm oil, ricebran oil, simarouba, soap nut, tumba, waste cooking oil and algae.

2.1. Algae oil (cyanobacteria)

Algae (micro and macro) oils are one of the best sources for synthesis of biodiesel. Micro algae are classified as diatoms (bacillar-iophyceae), green algae (chlorophyceae), golden brown (chryso-phyceae) and blue-green algae (cyano phyceae). Micro algae are the untapped resource with more than 25,000 species out of which only 15 are in use [47]. Microalgae may be the potential and economical source of biodiesel because of high yielding feedstock. Microalgae are an organism capable of photo-synthesis that is less than 2 mm in diameter. In microalgae the oil content can exceed 80% by weight of dry biomass and oil levels of 20–50% are common in any micro algae. Micro algae oils differ from most vegetable oils in being quite rich in polyunsaturated

Table 1
Potential non-edible oilseed plants in India.

Botanical name	Common name	Distribution	Potential (metric tons/ annum)	Oil (%)	Use	Refs.
Cyanobacteria	Algae	Few places in India	157.4–629.8	20–40	Human nutrition, animal feed, aquaculture, bio-fertilizer, source of polyunsaturated fatty acids, and recombinant proteins, etc.	[10,20,21]
Ricinus communis	Castor	All over India	790,000	46–55	Adhesives, coatings, soaps, lubricants, paints and dyes, etc.	[19]
Gossypium hirsutum	Cotton	All over India	851,000	18–25	Dairy cattle feeding, alternative bio-fuel source, etc.	[4,10,22–24]
Jatropha curcas	Jatropha	All over India	15,000	40–60	Biodiesel, mineral diesel substitute, to prevent and control erosion, to reclaim land, grown as a live fence, manufacture of candles, soap and cosmetics	[25,26]
Simmondsia chinesis	Jojoba	Few places in India	–	45–55	Cosmetics, skin-softener, pharmaceuticals, lubricants and synthesis of biodiesel	[27,28]
Pongamia pinnata	Karanja	Maharashtra, Karnataka, Assam	2,00,000	30–40	Tanning leather, soap, illuminating oil, lubricant, water-paint binder, and pesticide	[15,29,26]
Garcinia indica	Kokum	Western Ghats region, Andaman and Nicobar Islands, North-eastern region of India	55,000		Ideal feed stock for biodiesel	[26,30]
Linum Usitatissimum L.	Linseed	Few places in India	150,000	35–45	Textiles, oil crop, stem fibers, geotextiles, filters, absorbents, pulp and paper manufacturing, etc.	[13,27,31,32]
Madhuca indica	Mahua	Maharashtra, Gujarat, West Bengal, Karnataka, Orissa, Tamil Nadu, Kerala and West Bengal	5,20,000	35–40	Ointment, rheumatism, illumination, lighting, soaps, etc.	[26,33]
Moringa oleifera	Moringa	Himalayan regions of northwest India,	–	33–41	skin diseases, medicinal	[34,35]
Mesua ferrea	Nahor	Northeastern states of India	–	58–75	Production of biodiesel, wood as a railway crossties, heavy construction, boat building, mine props, and tool handles	[36,37]
Azadirachta indica	Neem	All over India	5,00,000	35 ± 45	Preservation of stored grains, Ayurvedic medicine, Unani, Homeopathic medicine, etc.	[26,38]
Erythea salvadorensis	Palm	–	< 70,000	20–21	Cosmetics, soaps, lubricant for biodiesel engine, diesel fuel substitute, transportation fuel	[4,39,40–42]
Oryza sativa	Rice bran	Andhra Pradesh, Orissa, Karnataka, West Bengal, Gujarat, Utter Pradesh	474,000	16–32	Non-edible vegetable oil, replacement for mineral diesel	[23,43]

Table 1 (*continued*)

Botanical name	Common name	Distribution	Potential (metric tons/ annum)	Oil (%)	Use	Refs.
Simarouba glauca	Simarouba	Gujarat, Maharashtra, Tamilnadu, Karnataka, Orissa and Andhra Pradesh	1.1–2.2 oil/ ha yr	55–65	Manufacture of soap, vegetable fat or margarine, lubricant, paint, polishes and pharmaceuticals	[44]
Sapindus mukorossi	Soapnut	Andhra Pradesh, Karnataka, Maharashtra, Delhi, Tamil Nadu	6.61	51.8	Medicinal, soap, surfactant, fabrics, bathing, and biodiesel production	[45]
Citrullus colocynthis	Tumba	Few places in India	21,000 tons	–	Medicinal	[27]
–	Waste cooking oil and animal fats	Restaurants and households, large food processing and service facilities	1,135,000	–	Biodiesel production	[10,46]

Table 2
Micro-algae oil.
Source: [20,21,49].

No.	Botanical name	Distribution	Biomass productivity (g/l yr)	Oil (%)	Use
1	*Botryoccocus braunii*	Britain, Poland, Romania, Spain, North America, Caribbean Island, Turkey, China, Australia and New Zealand	10.8	25–75	Biodiesel production
2	*Chlorella* sp.	Taiwan, Germany, Japan	900	28–32	Human nutrition, cosmetics, aquaculture, biofuel production
3	*Crypthecodinium cohnii*	Britain	3600	20	Nutritional supplements
4	Cylindrotheca sp.	Britain, Croatia, Spain, Canary Islands, North America, Korea, Australia and New Zealand		16–37	
5	*Dunaliella primolecta*	Balearic Islands, Romania, Spain, Egypt, Iran, Australia and New Zealand	32.4	23	Dietary supplement, anticancer agent
6	*Isochrysis* sp.	Britain, Spain	28.8–61.2	25–33	Used in animal nutrition
7	*Monallanthus salina*	Britain	28.8	>20	
8	*Nannochloris* sp.		61.2–183.6	20–35	Food additive, animal and fish feed, biofuel production
9	*Nannochloropsis* sp.	Germany	72	31–68	Nutritional supplement, aquaculture
10	*Neochloris oleoabundans*	South-west Asia		35–54	Cosmetics and biofuel production
12	*Phaeodactylum tricornutum*	France, Germany, North America	86.4	20–30	Nutritional supplement, aquaculture, fuel production

fatty acids with four or more double bonds [10,48]. Micro algae have long been recognized as potentially good sources for bio-fuel production because of their high oil content, rapid biomass production (double their biomass production within 24 h) and it is much faster and easier to grow (Table 2).

2.2. Castor oil (Ricinus communis)

Castor plant has the botanical name *R. communis* of the family Eurphorbiacae. Different varieties of castor seeds are available in the world but on the average, they contain about 46–55% oil by weight. Castor is originally a tree or shrub that can grow above 10 m high, reaching an age 4 years. At present, the cultivated varieties grow to a height of 60–120 cm in 1 year. Castor grows in the humid tropics to the sub-tropical dry zones (optimal precipitation 750–1000 mm, temperature 15–38 °C). The castor plant grows wild in large quantities in most tropical and sub-tropical countries at low cost and the plant is known to tolerate varying weather conditions. Specifically, castor plant requires a temperature between 15 and 38 °C with low humidity throughout the growing season in order to obtain maximum yields [19]. The oil of castor plant is viscous, pale yellow, non-volatile, non-drying oil with a bland taste and sometimes used as a purgative and the oil has a slight odor. Relative to other vegetable oils castor oil has a good shelf life and it does not turn rancid unless subjected to excessive heat and is a good source of raw material for varieties of other field like adhesives, coatings, soaps, lubricants, paints and dyes. India is the world's largest exporter of castor oil; the other

major producers are China and Brazil. The total world production of seeds is estimated at around 1 million tons and the oil extracted is about 500,000 tons, with a productivity of 470 kg of oil per hectare [50]. Castor seeds are poisonous to humans and animals because they contain ricin, ricinine and certain allergens that are toxic, and they cause abdominal pain, vomiting and diarrhea. Indeed, as little as 1 mg of ricin acid can kill an adult [19].

2.3. Cotton seed oil (*Gossypium hirsutum*)

Cottonseed plant has the botanical name *G. hirsutum*; it is a cooking oil extracted from the seeds of cotton plant of various species, mainly *G. hirsutum* and *Gossypium herbaceum*. The cotton seed has a similar structure to other oilseeds such as sunflower seed, and in processing the oil is extracted from the kernel, which is surrounded by a hard outer hull. Cotton seed oil has mild taste and appears generally clear with light golden color. In prepared food, cottonseed oil is a favorite staple for salad oil, mayonnaise, salad dressing and similar products because of its favor stability. The cotton seed oil undergoes intensive treatment after extraction to reduce the level of gossypol found in untreated cottonseed oil, the consumption of which may produce undesirable side-effects and it is relatively low cost feed stock for biodiesel production as cotton seeds are considered as by-products or waste [10].

Cottonseed oil described by scientists as being "naturally hydrogenated" because the saturated fatty acids it contains are the natural oleic, palmitic and stearic acids. When hydrogenated to a typical iodine value of about 80, for example, its fatty acid profile shifts to 50% monounsaturated, 21% polyunsaturated and 29% saturated, which are all well within current diet/health guidelines. Its fatty acid profile generally consists of 70% unsaturated fatty acids including 18% monounsaturated (oleic), 52% polyunsaturated (linoleic) and 26% saturated (primarily palmitic and stearic). The annual production of cotton seed oil is 4.6 mt per annum [22].

2.4. Jatropha oil (*Jatropha curcas L.*)

Jatropha plant has the botanical name *J. curcas*, and it is a multipurpose bush/small tree belonging to the family of Euphorbiaceae; it can grow under a wide variety of climatic conditions like severe heat, low rain fall, high rain fall, and is a small deciduous tree (up to 5 m) which originates all over the India. The seeds contain 30–35% oil by weight, which can easily be converted into bio-diesel meeting the standards. Recently Jatropha is being considered as one of the most promising potential oil source to produce biodiesel all over the world. In normal conditions the plant will fruit once a year, yielding 2–5 tons of dry seed/ha year, after 5 years, depending on the genetic variety, agro-climatical conditions and the management input. The seeds and oil are not edible due to the presence of toxins as phorbol esters, trypsin inhibitors, lectins and phytates. The fact that Jatropha oil cannot be used for nutritional purposes without detoxification makes its use as energy or fuel source very attractive. The seeds contain about 30% oil. The oil of *J. curcas* consists of both saturated (14.2% palmitic acid and 7.0% stearic acid) and unsaturated fatty acids (44.7% oleic acid and 32.8% linoleic acid). The plant is regarded as an oil plant with multiple attributes, uses and considerable potential. The plant can be used to prevent and/or control erosion, to reclaim land, grown as a live fence, especially to contain or exclude farm animals and can be planted as a commercial crop. The wood and fruit of Jatropha can be used for numerous purposes including fuel, and crop is traditionally used for medicinal purposes. The seeds of Jatropha contain viscous oil, which can be used in soap, cosmetics industry, preparing candles, as a diesel/paraffin substitute or extender. Pant et al. [25] showed that depending on the types of species and climatic conditions its oil content varies, but mainly on the altitude where it is grown. In developing countries like India it has been identified as the major source of biodiesel [10,25,51,52].

2.5. Jojoba oil (*Simmondsia chinensis*)

The seed of jojoba plant, which has botanical name *S. chinensis*, appears to be a promising scope for cultivation in arid and semi-arid areas, a perennial shrub that grows naturally in deserts. The Jojoba seed is nut shaped and is around 1–2 cm long, with red-brown to dark-brown color. The seed contains 45 and 55 wt% of Jojoba oil-wax, a golden liquid that can be obtained by cool pressing or solvent extraction. Unlike vegetable oils and animal fats, Jojoba oil is not a triglyceride but a mixture of long chain esters (97–98 wt%) of fatty acids and fatty alcohols. Jojoba oil-wax contains minor amounts of free fatty acids and alcohols, phytosterols, phospholipids and trace amounts of a triacylglycerol. The chemical structure of jojoba oil allows its use in many lubricating oil formulation and biodiesel production. Jojoba oil and its derivatives find applications in the fields of cosmetics, pharmaceuticals and lubricants. In the automotive industry, jojoba oil is a superior lubricant in high speed machinery, tool work and metal cutting [53,54].

2.6. Karanja seed (*Pongamia pinnata*)

Pongamia oil is a non-edible oil extracted from seeds of *P. pinnata* (L.) Pierre, family Fabaceae commonly known as 'Karach', 'Karanja' in Assam. In India the plant is distributed in Andhra Pradesh, Orissa, Bihar, Jharkhand, Chhatisgarh, Madhya Pradesh, Karnataka, Kerala, West Bengal and Maharashtra. Karanja is medium sized fast-growing ever green tree, 12–15 m height, branches spread into hemispherical crown of dense green leaves. It has a disagreeable odor and bitter taste. Pods are elliptical, 3–6 cm long and 2–3 cm wide, thick walled and usually contain single seed. Seeds are 10–20 mm long and light brown in color. Pongamia can survive in adverse conditions like draught, heat, frost, salinity, etc. The yield of kernels per tree is between 8 and 24 kg [55]. The freshly extracted Karanja oil is yellowish orange to brown and rapidly darkens on storage. The karanja seed kernel contains 27–39 wt% oil. The oil is extracted from the kernel by traditional expeller, which yields 24–26% oil. The oil contains toxic flavonoids such as karanjin and di-ketone pongamol as major lipid associates, which make the oil non-edible. The oil has been used mainly for leather tanning, lighting and to a smaller extent in soap making, medicine and lubricants. The main constraint to greater use of karanja oil in soaps is its color and odor, as well as the ineffectiveness of conventional refining, bleaching and deodorization in improving the quality of the oil [56]. Among the non-edible oilseeds karanja is one of the potential plants having annual production of 2 lakh tons of oilseed or 60,000 tons of oil, while only 6% is being utilized presently [10,15,17,29,57].

2.7. Linseed oil (*Linum usitatissimum*)

Linseed has the botanical name . *usitatissimum*, also known as flax seed oil, and is a clear to yellowish oil obtained from the dried ripe seeds of the flax plant. The linseed contains 35–45 wt% of oil. The oil is obtained by cold pressing, alkali refined, sun bleached, sun thickened and sometimes by solvent extraction method. Linseed oil is a so-called drying oil, which means that it hardens upon exposure to air; it is a mixture of various triglycerides that differ in terms of their fatty acid constituents. It is an edible oil, but due to its strong flavor and odor it is only a minor constituent of human nutrition, although it is marketed as a nutritional

supplement. Having a high content of unsaturated esters, due to its polymer-like properties, it has a significant applications in different fields, and it is used on its own or blended with other oils, resins and solvents as an impregnator and varnish in wood finishing, as a plasticizer and hardener in putty and in the manufacture of linoleum, and it is a common carrier as a pigment binder used in oil paints. The use of linseed oil was a significant step in the technology of oil painting. The annual production of linseed in India is 500 tons [13,27,31].

2.8. Mahua oil (Madhuca indica)

Mahua oil (*M. indica*) is obtained from the seeds of *M. indica* tree, which is found in most parts of India like (Maharashtra, Gujarat, Chhattisgarh, Karnataka, Orissa, Tamil Nadu, Kerala and West Bengal). Mahua seed contains about 35–40% of oil and the kernel constitutes about 70% oil. Each tree yields about 20–40 kg of seed per year. The unrefined but filtered crude mahua oil is greenish yellow in color [58]. It takes 8–15 years to mature fully and amature tree can bear fruits up to 60 years. It is the native of tropical-based deciduous forest of India. Mahua oil, flowers and its cake have some specific applications; the oil can be used in soap making and biodiesel production, the flowers of mahua that yield pure mahua oil are good feedstock for making ethanol at very low cost and the oil cake can be used in poultry feed. The mahua oil generally contains about 20% FFAs and in India it has an estimated annual production potential of 181,000 metric tons [10,59,60].

2.9. Moringa (Moringa oleifera)

Moringa is a single-genus family of oilseed trees with 14 known species, among these *M. oleifera* is the most widely known and utilized [61,62]. It is commonly known as the horse-radish or drumstick tree and is a native of the sub-Himalayan region of northwest India. The tree ranges in height from 5 to 12 m and the fruits (pods) are around 50 cm long. Fully mature, dry seeds are round or triangular in shape, the kernel surrounded by a light wooded shell with three papery wings; *M. oleifera* seeds contain between 33% and 41% oil [62]. Almost every part of the tree is of value for food. *Moringa oleifera* seed oil is pleasant tasting, is highly edible [63] and resembles olive oil in its fatty acid composition [64]. The seed oil contains all the main fatty acids found in olive oil and therefore can be used as a possible substitute to the expensive olive oil after some modifications, and seeds can be used as flocculant in water treatment. Almost all parts of the tree have been utilized within traditional medicine practices. The fast growing, drought-tolerant *M. oleifera* can tolerate poor soil, a wide rainfall range (25 to 300+ cm per year), and soil pH from 5.0 to 9.0 [62,65–67]. *M. oleifera* can be found in Andhra Pradesh, Tamilnadu and Karnataka. It has an annual production of 1.1–1.3 million tons of tender fruits from an area of 380 km^2.

2.10. Nahor (Mesua ferrea)

M. ferrea (commonly known as iron wood or nahor) is a tall tree reaching up to 100 feet tall, the base with a trunk up to 2 m in diameter. It has simple, narrow, oblong, dark green leaves 7–15 cm long, with a whitish underside; the emerging young leaves are red to yellowish pink and drooping. The flowers are 4–7.5 cm diameter, with four white petals and a center of numerous yellow stamens, trees, often tressed at the base with trunk diameters up to 3 feet and its timber is one of the hardest and heaviest. *M. ferrea* produces high oil content seeds (58–75%) and it contains both saturated and unsaturated fatty acids and triglycerides of linoleic, oleic, palmitic and stearic acids. It was also found that its oil contained essentially 74.6% unsaturated fatty acids comprising mainly of oleic acid (52.3%) and linoleic acid (22.3%) and 25.4% saturated fatty acids.

The seed oil is considered to be very useful in conditions like vata and skin diseases. Dried flowers are used for bleeding hemorrhoids and dysentery with mucus. Fresh flowers are useful remedy for itching, nausea, erysipelas, bleeding piles, metrorrhagea, menorrhagea, excessive thirst and sweating. Oil from the seeds is used for sores, scabies, wounds and rheumatism. In Assam *M. ferrea* seeds are produced annually more than 10 million kg. All the seeds are not utilized in production of biodiesel [36,37,68].

2.11. Neem (Azadirachta indica)

Neem (*A. indica*) is a fastest-growing tree that can reach a height of 15–20 m. It is evergreen, but in severe drought it may shed most or nearly all of its leaves. The branches are wide spread. Neem can be grown on very marginal soils that may be very rocky, shallow, dry orpan forming. The fairly dense crown is round or oval and may reach the diameter of 15–20 m in old, free-standing specimens. Very young leaves are reddish to purplish in color. The trunk is relatively short, straight and may reach a diameter of 1.2 m. The fruit is a smooth (glabrous) olive-like drupe that varies in shape from elongate oval to nearly roundish, and when ripe are 1.4–2.8 $\times$ 1.0–1.5 cm. The neem seed contains 35–45% of oil. The neem tree is native to India Neem oil, and is light to dark brown in color and bitter in taste. For neem seed oil, Azadirachtin is the main constituent and it varies from 300 to 2500 ppm depending on the extraction technology and quality of the crushed neem seeds.

Neem products have been observed advantageous to be anthelmintic, antifungal, antidiabetic, antibacterial, antiviral, contraceptive and sedative. Neem products are also used in selectively controlling pests in plants. It is considered a major component in Ayurvedic medicine and is particularly prescribed for skin disease. Neem gum is used as a bulking agent and for the preparation of special purpose food (for diabetics) and neem leaf paste is applied to the skin to treat acne. Neem oil has been found to be an effective mosquito repellent [10,26,38].

2.12. Palm (Elaeis guineensis)

Palm (*E. Guineensis*) oil is derived from the seed of the palm, and its trees are single-stemmed, and grow up to 20 m tall. The palm fruit is reddish, about the size of a large plum and grows in large bunches. Each fruit is made up of oily, fleshy outer layer with a single seed (the palm kernel), also rich in oil content 20–21%. When ripe, each bunch of fruit weighs 40–50 kg. Oil is extracted from both the pulp of the fruit (edible oil) and the kernel (palm kernel oil, used in foods and soap manufacturing). For every 100 kg of fruit bunches, typically 22 kg of palm oil and 1.6 kg of palm kernel oil can be extracted. The high oil yield of oil palm trees (as high as 7250 l per hectare per year) has made it a common cooking ingredient in south East Asia. Palm oil has been proved to be an efficient biodiesel source [10,39,69].

2.13. Rice bran oil

Rice bran is a brown layer present between rice and the outer husk of the paddy and it is a non-conventional, inexpensive, low-grade vegetable oil for biodiesel production. It is extracted from the germ and inner husk of rice. Depending on variety of rice and degree of milling, the bran contains 16–32 wt% of oil. Rice bran oil (RBO) is considered to be one of the most nutritious oils due to its favorable fatty acid composition and unique combination of naturally occurring biologically active antioxidant compounds.

Table 3
Physicochemical properties of the biodiesel feedstocks.

Chemical properties	Castor oil	Cotton seed	Jatropha	Jojoba	Karanja	Kokum	Linseed	Mahua	Moringa	Nahor	Refs.
(a)											
Unsaponifiable matter (w/w percent)	–	–	–	–	3.3	–	–	1	–	2.7	[33,37]
Specific gravity	0.96	0.9148 at 15 °C	0.912 at 15 °C	0.8635–0.8640 at 40 °C	0.882, at 15.5 °C	0.895 at 40 °C	0.931 – 0.938 at 15.5 °C	0.856, at 15 °C	0.907	0.92 at 30 °C	[26,33,36,37,72,73]
Kinematic viscosity at 30°C (mm^2/s)	29.7	33.5 at 38 °C	29.4	11.82 at 40 °C	27.8	–	22.2 at 40 °C	24.5 at 40 °C	43.4 at 38 °C	4.1 at 40 °C	[2,10,22,35,54,74,75,]
Calorific value (MJ/kg)	39.5	39.648	39.23	42.17	34.000	–	–	38.863	–	36	[23,36,54,72]
Iodine value (g I_2/100 g)	88.72	104.7	108.4	82–89	29.9	25–38	156.74	58–70	73	89.4	[2,10,35,37,74,76]
Acid value (mg KOH/g)	–	0.16	28	–	5.06	–	–	38	1.194	34	[2,36,73,77]
Saponification value (mg KOH/g)	191	198.5	200.8	92–167	188.5	187–192	187.6	190.5	199.7	190.6	[10,37,78]
Flash point (°C)	260	234	225	292	205	–	241	232	–	172	[2,10,23,36,72,75]
Aniline point (°C)	–	–	–	52.9	–	–	–	–	–	–	[79]
Cetane no.	42.3	54	61–63	–	60–61	65.6	34.6	56.61	56.66	54.6	[2,10,23,37,75,78]
Pour point (°C)	−32	258k	2	6	−3	–	−15	15	–	−1.2	[23,37,72,75,76,80]
Carbon residue (wt%)	0.21	0.42	0.44	–	0.71	–	< 0.01	0.42	–	–	[23,51,74,75,80]
Moisture content (wt%)	0.15-1.30	–	–	–	19%	–	–	1.6	7.9 ± 1	0.1	[29,36,67,77,81]

Chemical properties	Neem	Palm	Rice bran	Simarouba	Soap nut	Tumba	Waste cooking	Refs.
(b)								
Unsaponifiable matter (w/w percent)	–	–	–	0.40	–	–	–	[44]
Specific gravity	–	0.9180, at 15 °C	0.92, at 30°	–	–	0.924–0.927 at 28 °C	0.925	[26,43,82]
Kinematic viscosity at 30 °C (mm^2/s)	50.3	5.7	36.68 at 40 °C	–	–	–	36.4, at 40 °C	[10,16,83]
Calorific value (MJ/kg)	–	36.510	39.500	–	–	–		[23,83]
Iodine value (g I_2/100 g)	65–80	35–61	90–108	53.8	64.5	118–122	141.5	[10,16,44,76,78]
Acid value (mg KOH/g)	–	6.9	0.45	–	–	–	0.15	[2,7,42]
Saponification value (mg KOH/g)	209.66	208.6	201.27	191	195	172–174	188.2	[10,16,44,78]
Flash point (°C)	–	164	316	–	–	–	212	[16,43,83]
Cetane no.	57.83	62	50.1	59.32	59.77	–	49	[16,43,78,83]
Pour point (°C)	–	-31.7	1	–	–	–	11	[16,23,43]
Carbon residue (wt%)	–	–	0.6	–	–	–	0.46	[16,43,75]
Moisture content (wt%)	–	0.02	0.02	–	–	–	0.42	[7,16,42]

Table 4
Fatty acid composition of oils (wt%).

Fatty acids	Castor	Cotton seed	Jatropha	Jojoba	Karanja	Kokum	Lin seed	Mahuwa	Moringa	Refs.
(a)										
Myristic acid ($C_{14:0}$)	0	0.7	1.4	–	0	15.54	0	1	–	[10,30,37,74,84–86]
Palmitic acid ($C_{16:0}$)	1.1	28.7	15.6	1.08	14.1	2.5	5	17.8	7	[35,37,54,74,78,85,86]
Stearic acid ($C_{18:0}$)	3.1	0.9	9.7	–	10.9	56.4	2	14	4	[10,35,37,74,78,85,86]
Oieic acid ($C_{18:1}$)	4.9	13	40.8	7.22	56	39.4	18.9	46.3	78	[35,37,54,74,78,85,87]
Linoleic acid ($C_{18:2}$)	1.3	57.4	32.1	0.04	15	1.7	18.1	17.9	1	[35,37,54,74,78,85,87]
Linolenic acid ($C_{18:3}$)	0	0	0.2	–	3.6	–	55.1	<1	$<$ 1%	[10,35,74,76,78,87]
Eicosanoic acid ($C_{20:0}$)	0	0	0.4	0.23	2.1	15.79	0	3	4	[30,35,37,54,74,84–86]
Eicosenoic acid ($C_{20:1}$)	0.3	0	–	–.	2.4	6.17	–	–	–	[2,10,30,72,76,85]
Docosanoic acid ($C_{22:0}$)	–	0	0–0.2	0.29	1.9	0.01	0	4	4	[10,26,30,35,37,84,86]
Tetracosanoic acid ($C_{24:0}$)	–	0	14	–	2.4	–	0	–	–	[10,26,78,84–86]
Unaccounted for	89.6	0	1.4	14.2	4	7.42	0	–	–	[10,27,30,72,74,85,88]

Fatty acids	**Nahor**	**Neem**	**Palm**	**Rice bran**	**Sal**	**Simarouba**	**Soapnut**	**Tumba**	**WCO**	**Micro algae**	**Refs.**
(b)											
Myristic acid ($C_{14:0}$)	2.13	0.2–0.26	1	0.4–0.6	–	–	–	–	0.5		[13,36,78,84,85,86,89,90]
Palmitic acid ($C_{16:0}$)	15.3	13.6–16.2	42.6	11.7–16.5	4.5–8.6	19	4.67	13–14	20.4	12–1	[13,20,37,51,74,76,78,86]
Stearic acid ($C_{18:0}$)	12	14.4–24.1	4.4	1.7–2.5	34.2–44.8	14	1.45	9–10	4.8	1–2	[13,20,37,51,74,76,78,86]
Oieic acid ($C_{18:1}$)	53.2	49.1–61.9	40.5	39.2–43.7	34.2–44.8	63	52.64	26–28	52.9	58–60	[13,20,37,51,74,76,78,86]
Linoleic acid ($C_{18:2}$)	17	2.3–15.8	10.1	26.4–35.1	2.7	3	4.73	51–52	13.5	4–20	[13,20,37,51,74,76,78,86]
Linolenic acid ($C_{18:3}$)	–	–	0.2	0.6	–	–	1.94	–	0.8	14–30	[20,43,51,74,76,78,85,86,89]
Eicosanoic acid ($C_{20:0}$)	2	0.8–3.4	0	0.4–0.6	6.3–12.2	1	7.02	–	0.3	–	[13,20,37,51,78,86,89,90,91]
Eicosenoic acid ($C_{20:1}$)	–	–	0.1	–	–	–	23.85	0.4	0.3	–	[20,51,78,85,86,89,90,92]
Docosanoic acid ($C_{22:0}$)	0.5	–	0	–	–	–	1.45	0.8	0.07	–	[20,37,51,78,85,86,89,91,93]
Tetracosanoic acid ($C_{24:0}$)	–	–	0	0.4–0.9	–	–	0.47	0.3	0.04	–	[13,20,51,78,85,86,89,91,93]
Unaccounted for	–	–	1.1	–	–	–	–	–	–	–	[20,74,78,85,86,89,90]

Due to the presence of an active lipase in the bran, free fatty acid (FFA) content in RBO is much higher than other edible oils. Because of this reason, about 60–70% of the RBO production is non-edible. Thus, most of the rice bran is used as cattle feed in China. Rice bran oil (RBO) offers significant potential not only as a low-cost feedstock, but also as an alternative for biodiesel production in India and many other countries. The total potential amount of rice bran in India is about 474,000 tons/year [7,10,23,43].

2.14. Simarouba (Simarouba glauca)

Simarouba (*S. glauca* DC) is commonly known as paradise tree. Simarouba is a medium sized evergreen tree (height 7–15 m) with tap root system and cylindrical stem. It is a multipurpose tree capable of growing on the marginal waste land/dry land with degraded soils and can be adapted to a wide range of temperatures (10–45 °C) and altitudes up to 1000 m above sea level. The seed contains 50–65% oil that can be extracted by conventional method, and can be used for edible and non-edible purposes. Each well grown tree yields 15–30 kg nutlets per year equivalent to 2.5–5 kg oil. Simarouba is a unique tree and all its parts are useful in one way or the other and the oil is used in soap manufacturing industries, lubricant, paint and pharmaceuticals. Shells (endocarp) are used in hard board industry, dry fractionation of *S. glauca* is suitable for use as cocoa butter extender in chocolate products, and leaf litter makes good manure. Bark and leaf of simarouba contain triterpenes useful in curing amoebiasis, diarrhea and malaria.

Cultivation of simarouba was introduced in India by National Bureau of Plant Genetic Resources as a potential source of vegetable oil during late sixties in sub-humid climate of Orissa. Later in seventies, the cultivation of simarouba spread to semi arid, dry and saline land areas of other Indian states like Gujarat, Maharashtra, Tamilnadu, Karnataka and Andhra Pradesh [40,44,59,70].

2.15. Soap nut (Sapindus mukorossi)

Soap nut (*S. mukorossi*) is a fruit of the soap nut tree and the plant grows very well in deep loamy soils and leached soils, so cultivation of soap nut in such soil avoids potential soil erosion. The kernels from the shells for oil extraction can be separated and then cold pressed, approximately 1.5 g of oil is recovered from 5 g of kernels (30% oil content). The soap nut tree can be used for multiple applications such as rural building construction, oil and sugar presses. Also it is used in a commercial cleanser and medical applications based on its usages including antidermatophytic, antitussive and antihelmintic activities [71]. Generally these are found in tropical and sub-tropical climate areas in various parts of the world. Two main varieties (*S. mukorossi* and *S. trifoliatus*) are widely available in India. The oil content in *S. trifoliatus*, which is very similar to *S. mukorossi* seed kernels, is on average 51.8% of seed weight. The oil from soap nut has been considered as non-edible oil having significant potential for biodiesel production. Among the non-edible oilseeds, soapnut is one of the potential plants having annual production of 6000 kg oilseeds [45,51].

2.16. Waste oils

Waste oil is defined as any oil based on edible, non-edible, petroleum or synthetic oils, through use or handling it becomes unsuitable for its original purpose due to the presence of impurities or loss of original properties. Used frying oil (UFO) is not suitable for human consumption but it is one of the good feedstock for biodiesel production. Every day in the world, there is a

Table 5
Specification of biodiesel for US, European countries and India.
Source: [2,85].

Sl. no.	Properties	EN 14214-2008	IS 15607-2005	ASTM D 6751-09
1.	Ester content, %(m/m)	96.5	96.5	–
2.	Density at 15 °C (kg/m^3)	860–900	860–900	–
3.	Viscosity at 40 °C (mm^2/s)	3.5–5		1.9–6 °C (cSt)
4.	Flash point, °C (min)	> 101 °C	120	130–403 K
5.	Sulfur content (mg/kg)	10	50 ppm max	0.05 max wt %
6.	Tar remnant (at 10% distillation remnant), %(m/m)	0.3	–	
7.	Cetane number	51	51	47 min
8.	Sulfated ash content, %(m/m)	0.02	0.02	0.02 max wt%
9.	Water and sediment content (mg/kg)	500	500 ppm max	0.05 max vol%
10.	Total contamination (mg/kg)	24	24 ppm	–
11.	Copper band corrosion (3 h at 50 °C), rating	Class 1	1 max	–
12.	Oxidation stability at 110 °C (h)	6	6	–
13.	Acid value, mg KOH/g	0.5	0.5	0.08 max
14.	Iodine value	120	To report	–
15.	Linoleic acid methylester, % (m/m)	12	–	–
16.	Polyunsaturated (P4 double bonds) methylester, %(m/m)	1	–	–
17.	Methanol content, %(m/m)	0.2	0.2	–
18.	Monoglyceride content, %(m/m)	0.8	–	–
19.	Diglyceride content, %(m/m)	0.2	–	–
20.	Triglyceride content, %(m/m)	0.2	–	–
21.	Free glycerol %(m/m)	0.02	0.02	0.02 max
22.	Total glycerin, %(m/m)	0.25 max	0.25	0.24 max
23.	Alkali metals (Na+K) (mg/kg)	5	–	–
24.	Phosphorus content (mg/kg)	10	10	0.001 max
25.	Kinematic viscosity (at 313 K) (mm^2/s)	3.5–5	2.5–6	1.9–6.0 mm^2/s
26.	Copper strip corrosion	Class 1		No 3 max
27.	Carbon residue, % by mass	0.3 max	0.05 max	0.05 max mass %
28.	Acid value, mg KOH/g	0.5 max	–	0.8 max
29	Cloud point (°C)	0.5,257	–	Report customer
30	Distillation T90 AET (°C)	–	–	360 max
31	Neutralization value (mg, KOH/gm)		–	0.8 max
32	Phosphorus (mass%)	10 mg/kg, 257	–	0.001 max

large amount of waste lipids generated from restaurants, food processing industries, fast food shops and households. Disposal of waste cooking oil and fats becomes a serious environmental problem in many of the countries around the world. This environmental problem could be solved by proper utilization and management of waste cooking oil as a diesel fuel. Due to the high cost of crude and refined vegetable oils for the production of biodiesel, cheaper animal fats and used cooking oils available from restaurants and households are attracting attention as an alternative feed stock for biodiesel production. Its usage significantly reduces the cost of biodiesel production. However, the quality of UFO may cause concern because its physical and chemical properties depend on the contents of fresh cooking oil and UFO may contain lots of undesired impurity, such as water, free fatty acids [2,10].

The physicochemical properties of biodiesel feed stocks and their fatty acid composition are listed in Tables 3 and 4, respectively. The biodiesel produced from either of this feedstock should meet the biodiesel specifications as listed in Table 5. In the subsequent sections different transesterification processes and technical aspects have been discussed in detail [94,95].

Table 6
Homogeneous base/alkali catalyzed transesterification.

Oil	Catalyst	Catalyst amount (%)	Alcohol	Oil to alcohol molar ratio	Reaction conditions	Ester yield (%)	Refs.
Karanja oil	KOH	1	Methanol	1:6	65 °C, 2 h, 360 rpm	98	[15]
Rapeseed oil	KOH	1	Methanol	1:6	65 °C, 2 h, 600 rpm	96	[103]
Sunflower oil	NaOH	1	Methanol	1:6	60 °C, 2 h, 600 rpm	97	[104]
Used frying oil (UFO)	NaOH	1.1	Methanol	1:7.5	70 °C, 30 min	85.3	[105]
	KOH	1.5					
Madhuca indica	KOH	0.7	Methanol	1:6	1 h	–	[77]
Rubber seed	NaOH	0.5	Methanol	1:9	45 ± 5 °C, 30 min	–	[106]
Used frying oil (UFO)	NaOH	1.1	Methanol	1:7	70 °C, 20 min	94.6	[105]
Tobacco	KOH	1	Methanol	1:6	30 min	–	[105]
Sunflower	KOH	–	Methanol	1:6	25 °C	90	[107]
Peanut	NaOH	–	Methanol	–	50 °C	90	[108]
Waste cooking oil (WCO)	NaOH	1.1	Methanol	1:7	60 °C, 33 min	88.8	[5]
Waste cooking oil (WCO)	KOH	6	Methanol	1:9	87 °C, 2 h	87	[5]
Soybean	KOH	0.8	–	–	40 °C, 1 h	95	[109]
Sunflower	NaOH	1	–	–	70 °C, 2 h	97	[89]
Cottonseed	$NaOCH_3$	0.75	–	–	75 °C, 1.5 h	97	[110]
Rice bran	$NaOCH_3$	0.88	–	–	65 °C, 1 h	83.3	[111]
Palm	NaOH	1	–	–	70 °C, 0.5 h	95	[112]
Jatropha	KOH	1	–	–	75 °C, 1 h	97.6	[113]
Waste frying	KOH	1.2	–	–	70 °C, 2 h	95.8	[89]
Karanja	KOH	–	Methanol tetrahydro-furan (THF)	1:10	60 °C	92	[114]
Jojoba	KOH	1.35	Methanol	–	25 °C	83.5	[53]
Rapeseed	KOH	–	$NaOCH_3$	–	2 h	98	[115]
Sunflower	KOH	0.28	–	1:3	70 °C	96	[116]
Palm	KOH	1	–	1:6	50 °C, 1 h	75	[117]
Jatropha	NaOH	3.3	–	–	65 °C, 2 h	55	[118]
Palm	$NaOCH_3$	–	Methanol	1:13	60 °C, 15 s	96, 99	[119]
Jatropha	NaOH	1	Methanol	1:5	60 °C, 90 min	98	[120]
Jatropha	NaOH	1.4	Methanol	1:4.2	65 °C, 120 min	90	[120]
Jatropha	KOH	2	Methanol	1:9	60 °C, 120 min	95	[120]
Jatropha	KOH	1	Methanol	1:6	65 °C, 60 min	99	[120]
Jatropha	KOH	1.1	Methanol	1:11	66 °C, 120 min	93	[120]
Jatropha	KOH	0.55	Methanol	1:5	60 °C, 24 min	99	[120]
Jatropha	Alumina loaded with potassium nitrate	6	Methanol	1:12	70 °C, 360 min	84	[120]
Neem oil	NaOH	0.7	Methanol	1:6	60 °C, 6.5 h	94	[121]
Neem oil	NaOH	0.7	Ethanol	1:10	80 °C, 8 h	88	[121]
Palm olein	Boiler ash (BA)	3	Methanol	1:15	60 °C, 0.5h	90	[122]
Waste cooking oil	NaOH	1	Methanol	7:3	65, 50 °C, 6 h	90.6	[123]
Safflower oil	NaOH	1	Methanol	1:10	–, 6 min	98.4	[124]
Castor oil	NaOH	1, 1.5, 2	Ethanol	1:6, 9	60 °C, 1 h	–	[125]
Palm oil	NaOH, KOH	1	Methanol	1:6	60 °C, 1 h	–	[126]
Refined sunflower oil	NaOH, KOH, CH_3ONa, CH_3OK	0.45–1.5	Ethanol	1:5	20–70 °C, 1–3 h	< 90	[127]
High oleic sunflower oil	Sodium ethoxide	1	Ethanol	1:3, 1:6, 1:9	30–80°C, 3–60 min	46.3–99.6	[128]
Soybean oil	KOH	0.5,1,1.5	Methanol	1:3, 1:6, 1:9, 1:12	50–110 °C, 2–50 min	99	[129]
Waste cooking oil	KOH	1	Methanol	1:6	70 °C, 1 h	98.2	[130]

3. Biodiesel production process

Transesterification reaction is important in the production of biodiesel and is carried out by the reaction of fats or oils with alcohols. Generally two mostly used methods for transesterification reaction are catalytic and non-catalytic transesterification and other methods such as supercritical processes, microwave and ultrasound systems. Transesterification reaction can be catalyzed by both homogeneous (alkalies and acids) and heterogeneous catalysts. Homogeneous catalysts show greater performance toward transesterification to obtain bio-diesel when the free fatty acid content is $<1\%$ [10]. Homogeneous catalysts have certain disadvantages in their application such as expensive separation of the homogeneous catalyst from the reaction mixture, generation of large amount of waste water during separation and cleaning of catalyst and the products, formation of unwanted by-product (i.e. soap) by reaction of the FFA [96].

Heterogeneous catalysts have been tried to overcome the drawbacks of the homogeneous catalysts. Heterogeneous catalysts are promising for the transesterification reaction of vegetable oils when the free fatty acid (FFA) content is $>1\%$ and can be separated more easily from reaction products. Uses of these catalysts avoid the undesired saponification reactions and also facilitate the transesterification of vegetable oils or animal fats with high contents of FFAs, for example, deep-frying oils from restaurants and food processing. They also show less corrosive nature which makes them better alternative for homogeneous catalyst. Heterogeneous catalysts are environmentally benign, could be operated in continuous processes and more over they can be reused and regenerated. The transesterification process for biodiesel production is conventionally achieved using homogeneous and heterogeneous acid or base catalysts [84,97].

In general, a catalyst is used to initiate the esterification reaction for making biodiesel. The catalyst increases the reaction rate and enhances the solubility of alcohol. Acid-catalyzed reactions (i.e. H_2SO_4, H_3PO_4) are used to convert FFAs to esters, or soaps to esters as a pretreatment step for high FFA feedstocks and these are characterized by slow reaction rate and high ratio of alcohol [98]. A high conversion efficiency with acid-catalyzed transesterification can be achieved by increasing the molar ratio of alcohol to oil, reaction temperature, catalyst loading and the reaction time [99,100].

Base-catalyzed (i.e. NaOH, KOH, and NaMeO, CH_3ONa) process is relatively fast but is affected by water content and FFAs of oils or fats. FFAs can react with base catalysts to form soap and water. Formation of soap not only lowers the yield of alkyl esters but also increases the difficulty in the separation of biodiesel, glycerol and also in the water washing because of the formation of emulsions. From the literature it is observed that mostly methoxide catalysts give higher yields than hydroxide catalysts, and potassium-based catalysts give better biodiesel yield than sodium-based catalysts [5].

The following section gives an overview of various acid, base catalytic (homogeneous, heterogeneous) and non-catalytic processes reported so far for biodiesel production.

3.1. Homogeneous alkali-catalyzed transesterification

Transesterification reaction can be catalyzed by both homogeneous alkali and acid catalysts. The most commonly used alkali

Table 7
Homogeneous acid catalyzed transesterification canola oil.

Oil	Catalyst	Catalyst amount (%)	Alcohol	Oil to alcohol molar ratio	Reaction conditions	Ester yield (%)	Refs.
Microalgae	H_2SO_4	–	Methanol	1:56	30 °C	60	[133]
Mahua	H_2SO_4	0.32	–	1.24 v/v	60 °C, 1 h	98	[77]
Rice bran		–	Methanol	1:10	60 °C	<96	[134]
Rubber seed	H_2SO_4	0.5	Methanol	1:6	45 ± 5 °C, 30 min	–	[106]
Soybean	H_2SO_4	3	n-Butanol	1:1	120 °C, 1 h	–	[135]
Soybean	H_2SO_4	1	Methanol, ethanol, butanol	1:30	65 °C, 50 h	–	[136,137]
					78 °C, 18 h	–	
					117 °C, 3 h	–	
Tobacco	H_2SO_4	1	Methanol	1:18	60 ± 0.1°C, 25 min	91	[138]
Waste cooking oil (WCO)	H_2SO_4	4	Methanol	1:20	95 °C, 1 h	–	[102]
Waste cooking oil (WCO)	H_2SO_4	4	Methanol	1:20	95 °C, 20 h	≥ 90	[5]
Waste cooking oil (WCO)	H_2SO_4	1	Methanol	1:30	65 °C, 69 h	99	[5]
Soybean oil	H_2SO_4	5	Methanol	1:6, 1:12, 1:24,	15, 25, 45, 65°C	–	[139]
Canola oil	Lewis acids ($AlCl_3$ or $ZnCl_2$)	1	Methanol	1:24	110 °C, 18 h	98	[140]
Refined soybean oil	Trifluoroacetic acid	2	Methanol	1:20	120 °C, 5 h	98.4	[141]
Waste cooking oil (WCO)	H_2SO_4	4	Methanol	1:20	95 °C, 20 h	>90	[142]
Waste cooking oil (WCO)	H_2SO_4	41.8	Methanol	1:245	70 °C, 4 h	99	[142]
Corn oil	p-Toluenesulfonic acid (PTSA), benzenesulfonic acid, H_2SO_4	1–4	Methanol	10–3	40, 60, 80 °C, 20 min	90.2–97.1	[143]
Canola oil	H_2SO_4, benzenesulfonic acid, p-toluenesulfonic acid or 2,4-dimethylbenzenesulfonic acid	1–5	Methanol	1:15	60 °C, 30 min	21–99.9	[144]

catalysts are NaOH, CH_3ONa and KOH [100]. The major advantages of using these catalysts (i.e. alkali catalysts) in the industries are: widely available, economical and competent to catalyze the reaction at low temperature and atmospheric pressure which leads to high conversion in a minimal time [101]. Only limitation of this catalyst is it can be used for refined vegetable oil with less than 0.5 wt% FFA or acid value less than 1 mg KOH/g [102].

Thus, for the feedstock with an average FFA content of more than 6 wt%, base catalyst is definitely not suitable [101]. The base-catalyzed reaction is reported to be very sensitive to the purity of the reactant. For identical experimental conditions compared to waste frying oils ($\sim$92%), virgin oil yields higher ester conversion ($\sim$97%) [89]. If the FFA content in the oil reaches higher value, for example, 3%. It has been found that the alkaline-catalyzed transesterification process is not suitable to produce esters. In order to prevent saponification during the reaction, FFA and water content of the feed must be below 0.5 and 0.05 wt%, respectively. Because of these limitations, only pure vegetable oil feeds are appropriate for alkali-catalyzed transesterification without extensive pre-treatment [17]. Table 6 shows the work carried out for bio-diesel production from various feedstocks under different conditions using homogeneous alkali catalysts.

3.2. *Homogeneous acid-catalyzed transesterification*

The transesterification process is catalyzed by H_2SO_4, HCl, BF_3, H_3PO_4, and organic sulfonic acids. Advantages of acid-catalyzed transesterification process are: directly it produces bio-diesel from low-cost lipid feedstocks, such as used cooking oil and greases, commonly have FFAs levels of $>6\%$ and insensitive to the FFAs content in the feedstock [100]. Since liquid base-catalyzed transesterification process poses lot of problems especially for oil or fat with high FFAs concentration, therefore liquid acid catalysts are proposed in order to overcome the limitations. However, the performance of the acid catalyst is not strongly affected by the presence of FFAs in the feedstock. In addition, economic analysis has proven that acid-catalyzed process, being a one-step process, is more economical than the base-catalyzed process which requires an extra step to convert FFA to methyl esters [131]. In general, acid-catalyzed reactions are performed at high alcohol-to-oil molar ratios, low-to-moderate temperatures and pressures, high acid catalyst concentrations, slow reaction rate, and therefore, it is not a popular choice for commercial applications [102,132]. Table 7 shows the widely used homogeneous acid-catalysts for transesterification reaction from various feedstocks. Freedman et al. [137] during their study reported 99% oil conversion using 1 mol% of H_2SO_4 and methanol to oil ratio 30:1 for 69 h reaction time. It shows that acid-catalyzed transesterification process requires more severe reaction conditions (such as longer reaction time, high alcohol ratio) than base-catalyzed reaction. Similarly, preliminary study to compare the transesterification of soybean oil with different alcohols such as methanol, ethanol and butanol with 6:1 and 20:1 M ratio in the presence of 1% concentrated sulfuric acid at 3 and 18 h, respectively, indicates unsatisfactory conversions to esters. However, 30:1 M ratio resulted in a high conversion to the methyl ester. Although it is insensitive to FFAs in the feedstock, homogeneous acid-catalyzed transesterification has been largely ignored mainly because of its relatively slower reaction rate.

Table 8
Heterogeneous acid catalyzed transesterification.

Oil	Catalyst	Catalyst amount (%)	Alcohol	Oil to alcohol molar ratio	Reaction conditions	Ester yield (%)	Refs.
Cottonseed	TiO_2/SO_4^{2-}	2	–	1:12	230 °C, 8 h	90	[147]
Jatropha	KNO_3/Al_2O_3	6	Methanol	1:12	70 °C, 6 h	84	[148]
Pongamia pinnata	ZnO	–	Methanol	1:10	105 °C, 1.5 h	–	[149]
Palm kernel oil, coconut oil	ZrO_2/SO_4^{2-}	1	–	1:6	200 °C, 1 h	90.3, 86.3 ME	[145]
Simaruba	Eu_2O_3/Al_2O_3	6.75	Methanol	1:5–6	70 °C, 8 h	63	[150]
Soybean	S-ZrO_2 Sulfated Zirconia	5	Methanol	1:20	120 °C, 1 h	98.6	[151]
Soybean	ZnO/I_2	Zn–5, I_2-2.5	Methanol	1:42	65 °C, 26 h	–	[152]
Soybean	$Al_2O_3/ZrO_2/WO_3$	4	Methanol	1:40	250 °C, 20 h	90	[153]
Soybean	SBA-15-SO_3H–P123	10	Methanol	1:20	75 °C, 20 h	85	[154]
Sunflower	ZrO_2/WO_3^{2-}	15	–	1:20	200 °C, 5 h	–	[155]
Waste cooking oil (WCO)	(ZS/Si) Zinc stearate immobilized on silica gel	3	Methanol	1:18	200 °C, 10 h	98	[132]
Waste Cooking Oil (WCO)	Zeolite Y (Y 756)	–	Methanol	1:6	460 °C, 0.37 h	26.6	[5]
Waste cooking oil (WCO)					$H_3PW_{12}O_{40}.6H_2O(PW_{12})$	3.7	Methanol
1:70	65 °C, 14 h	87	[5]				
Waste cooking oil (WCO)	$SO_4^{2-}/SnO_2–SiO_2$	3	Methanol	1:15	150 °C, 3 h	92.3	[5]
Waste cooking oil (WCO)	TiO_2–MgO	–	Methanol	1:30	150 °C, 6 h	92.3	[156]
Cotton seed oil	SO_4^{2-}/ZrO_2	2	Methanol	1:12	230 °C, –	>90	[157]
Palm kernal oil	SO_4/SnO_2	3	Methanol	1:6	200 °C, –	80.6	[157]
Soybean oil	Silica-bonded N-propyl sulfamic acid	7.5	Methanol	1:20	149.85 °C, 60 h	90.5 con	[158]
Pistacia chinensis oil	CaO–CeO_2 mixed-oxide	9	Methanol	1:30	100 °C, 6 h	91	[159]
Rapeseed oil	Styrene–divinylbenzene macroporous	10	Methanol	–	100–140 °C, 4 h	16.5–55	[160]

3.3. Heterogeneous acid-catalyzed transesterification

Heterogeneous acid-catalyzed transesterification reactions have the strong potential to replace liquid acid catalyst [132]. Because of its obvious advantages biodiesel research is focused on exploring new and sustainable solid acid catalysts for transesterification reaction. The solid acid catalysts are insensitive to FFA content, esterification and transesterification occur simultaneously, avoids the washing step of biodiesel, separation of the catalyst from the reaction medium is easy, low product contamination, easy regeneration, recycling of catalyst and reduces corrosion problem even with the presence of acid species [100,145,146]. However, limitations of slow reaction rate and possible undesirable side reactions, research on direct use of solid acid catalyst for biodiesel production has not been widely explored. The ideal solid acid catalyst for transesterification reaction should have characteristics such as interconnected system of large pores, moderate to high concentration of strong acid sites and hydrophobic surface [100]. Satoshi et al. [108] compared the transesterification of soybean oil with methanol to fatty acid ester over the solid super acid catalysts such as tungstated zirconia–alumina (WZA), sulfated zirconia–alumina (SZA) and sulfated tin oxide (STO). The reaction was monitored in a fixed bed reactor under atmospheric pressure at 200–300 °C. It was reported that WZA is a promising solid acid catalyst for the production of bio-diesel from soybean oil (conversion over 90%). Table 8 details the work carried out for bio-diesel production from various feedstocks under different conditions using heterogeneous solid acid catalysts.

3.4. Heterogeneous alkali-catalyzed transesterification

The heterogeneous catalyst process is expected to be an effective biodiesel production process with low cost and minimal environmental impact because of the possibility of simplifying the production and purification processes under mild conditions. Therefore, many heterogeneous alkali catalysts for the transesterification of oils have been developed, such as basic zeolites, alkaline earthmetal oxides and hydrotalcites. The solid base catalysts are easily regenerated and have a less corrosive nature, leading to safer, cheaper and more environment-friendly operations. However, they can be directly used as catalysts to prepare biodiesel when the FFA content (in a raw material) is not more

Table 9
Heterogeneous base catalyzed transesterification.

Oil	Catalyst	Catalyst amount (%)	Alcohol	Oil to alcohol molar ratio	Reaction conditions	Ester yield (%)	Refs.
Jatropha	KNO_3/Al_2O_3	6	Methanol	1:12	70 °C, 6 h	–	[148]
Karanja	Li/Cao	2	Methanol	1:12	65 °C, 8 h	94.8	[100]
Palm	KF/Al_2O_3	4	Methanol	1:12	65 °C, 3 h	90	[162]
Pongamia Pinnata	Hb–Zeolite	–	Methanol	1:10	393 K, 24 h	–	[163]
Rapeseed	KF/Eu_2O_3	3	Methanol	1:12	65 °C, 1 h	–	[164]
Sunflower	(Magnesium–lanthanum–mixed oxide	5	Methanol	1:53	65 °C, 30 min	100	[165]
Sunflower	$Ca(C_2H_3O_2)_2$/SBA-15	1	Methanol	1:12	60 °C, 5 h	–	[166]
Sunflower	CaO	3	–	1:13	60 °C, 100 min	–	[167]
Soybean	KNO_3/Al_2O_3	6.5	Methanol	1:15	65 °C, 7 h	–	[168]
Soybean	Eu_2O_3/Al_2O_3	10	Methanol	1:6	70 °C, 8 h	–	[150]
Soybean	KI/Al_2O_3	2.5	Methanol	1:15	65 °C, 8 h	–	[169]
Soybean	Mg–Al hydrotalcites	7.5	Methanol	1:15	65 °C, 9 h	–	[170]
Soybean	KOH/NaX zeolite	3	Methanol	1:10	65 °C, 8 h	–	[171]
Soybean	Nano MgO	3	–	1:36	250 °C, 10 min	–	[172]
Soybean	CaO	8	–	1:12	65 °C, 3 h	–	[173]
Soybean	SrO	3	–	1:12	65 °C, 30 min	–	[174]
Soybean	ZnO/Ba	6	Methanol	1:12	65 °C, 1 h	–	[175]
Soybean	(Calcium ethoxide) $Ca(OCH_2CH_3)_2$	3	Ethanol	1:12	75 °C, 3 h	91.8	[176]
Waste cooking oil	K_3PO_4	4	Methanol	1:6	60 °C, 2 h	97.3	[5]
Jatropha curcas oil	CaMgO, CaZnO	–	Methanol	1:15	65 °C, 6 h	80 conversion	[156]
Rapeseed oil	KF/Eu_2O_3	–	Methanol	1:12	Methanol reflux, temperature, 1 h	92.5 conversion	[156]
Soybean oil	$Ca(OCH_2CH_3)_2$	–	Methanol	1:12	65 °C, 1.5 h	95	[156]
Soybean oil	$Eu(NO_3)_3/Al_2O_3$	–	Methanol	1:6	70 °C, 6 h	63 conversion	[156]
Soybean oil	Lithium-doped ZnO	–	Methanol	1:12	65 °C, 3 h	96.3 conversion	[156]
Soybean oil	KF/ZnO	–	Methanol	1:10	65 °C, 9h	87 conversion	[156]
Jatropha oil	Na doped SiO_2	–	Methanol	1:15	65 °C, 45h	99 conversion	[156]
Soybean oil	MgO	–	Methanol	–	250^{O}C, 10 min	< 99%	[177]
Soybean oil	MgO	–	Methanol	–	200^{O}C, 60 min	< 98%	[177]
Jatropha	CaO	–	Methanol	–	70 °C, 2.5 h	< 93%	[177]
Tributyrin	CaO	–	Methanol	–	60 °C, 2 h	< 95%	[177]
Tributyrin	Li–CaO	–	Methanol	–	60 °C, 10 min	< 100%	[177]
Karanja	Li-CaO	–	Methanol	–	65 °C, 8 h	< 94.9%	[177]
Canola oil	Na, K, LieBaO	–	Methanol	–	50 °C, 4 h	< 97.5%	[177]
Tributyrin	MgOeCaO	–	Methanol	–	60 °C, 3 h	< 60%	[177]
Palm oil	CaO/Al_2O_3	–	Methanol	–	65 °C, 3 h	< 95%	[177]
Waste frying oil	Mg-AL HTs	–	Methanol	–	140 °C, 6 h	< 100%	[177]
Rapeseed oil	Mg-AL HTs	–	Methanol	–	65 °C, 3 h	< 94%	[177]

than or equal to 1. The use of heterogeneous catalysts to replace homogeneous catalyst can be expected to eliminate the problems associated with homogeneous catalysts [161]. A research group reported that the calcinations of pulverized limestone such as $CaCO_3$ at 900 °C for 1.5 h give CaO, which can be used as a catalyst for the transesterification of soybean oil. The yield of fatty acid methyl esters was 93% after 1 h reaction time at methanol:oil ratio of 12:1 under reflux condition. On the other hand when waste cooking oil with FFA content of 2.6% was used under identical reaction condition the yield dropped to 66% (Table 9). Further they indentified the formation of soluble substance such as calcium diglyceroxide during the transesterification reaction. Thus, an extra purification step such as ion-exchange resin is needed to remove the soluble content in the biodiesel. Another group used alkaline earth metal oxides especially calcium oxide; CaO has attracted much attention due to its relatively high basic

Table 10
Enzyme catalyzed transesterification.

Oil	Source of enzyme	Amount of enzyme used	Solvent	Oil to alcohol molar ratio	Reaction conditions	Ester conversion (%)	Refs.
Cotton seed	*Candida antartica*	1.6	t-Butanol	1:4	50 °C,24 h	95	[180]
Sunflower	*Candida antartica*	3	–	–	318 K, 50 h	–	[181]
Sunflower oil	*Pseudomonas fluorescens*	10	Hexane	1:4.5	40 °C,48 h	91	[182]
Soybean	*Candida antartica*	4	–	–	303 K, 48 h	–	[183]
Soybean	*Thermomyces lanuginosus*	15	–	1:7.5	31.5 °C,7 h	96	[184]
Jatropha	*Chromobactrium viscosum*	10	–	1:4	40 °C,10 h	92	[185]
Palm	*Pseudomonas fluorescens*	20	–	1:18	58 °C, < 24 h	98	[186]
Jatropha	*Candida antartica*	10	–	1:11	40 °C, 12 h	91.3	[187]
Rapeseed	*Candida antartica*	3	t-Butanol	1:4	35 °C, 12 h	95	[188]
Rapeseed	*Candida antartica*	5	–	–	313 K, 24 h	–	[189]
Jatropha	*Pseudomonas cepacia*	5	–	–	323 K, 8 h	–	[190]
Rapeseed	*C. rugosa*	–	–	–	–	97	[191]
Sunflower	*M. miehei (Lipozyme)*	–	–	–	–	83	[192]
Fish	*C. anturctica*	–	–	–	–	100	[193]
Palm kernel	*I. cepuciu (Lipase PS-30)*	–	–	–	–	15, 72	[194]
Palm	*Rhizopus oryzae*	–	Water	–	–	55 (w/w)	[195]
Sunflower Soybean	Novozyme 435	–	–	–	–	97	[97]
Cotton	Novozyme 435	–	–	–	–	97	[97]
Jatropha	Novozyme 435	–	–	–	–	91.3	[97]
Waste cooling oil (WCO)	*Pseudomonas cepacia* (PS30)	13.7	–	1:6.6	38.4 °C, 2.5 h	–	[5]
Waste cooling oil (WCO)	*Candida antartica* (Novozyme 435)	4	–	1:3	30 °C, 50 h	90.4	[5]
Waste cooling oil (WCO)	*Bicillus subtilis*	3	–	1:1	40 °C, 72 h	–	[5]
Waste cooling oil (WCO)	*Rhizopus oryzae*	30	–	1:4	40 °C, 30 h	–	[5]
Waste cooling oil (WCO)	Immobilized pencillium	–	–	1:1	35 °C, 7 h	–	[5]
Castro oil	*Candida antartica* (Novozyme 435)	20	Ethanol	1:10	65 °C, 6 h	81.4	[196]
Soybean oil deodorizer distillate	*Candida antartica* (Novozyme 435)	3	Ethanol	1:2	50 °C, 1.5 h	83.5	[196]
Sunflower oil	*Candida antartica* (Novozyme 435)	30	Ethanol	1:5	45 °C, 12 h	27	[196]
Palm oil	*Candida antartica* (Novozyme 435)	30	Ethanol	1:5	45 °C, 10 h	75	[196]
Groundnut oil	*Candida antartica* (Novozyme 435)	30	Ethanol	1:5	45 °C, 10 h	75	[196]
Pongamia piñata oil	*Candida antartica* (Novozyme 435)	30	Ethanol	1:5	45 °C, 10 h	50	[196]
Jatropha curcas oil	*Candida antartica* (Novozyme 435)	30	Ethanol	1:5	45 °C, 10 h	55	[196]
Castro oil	*Candida antartica* (Novozyme 435)	20	Ethanol	1:5	45 °C, 12 h	37	[196]
Jatropha curcas oil	*Chromobacterium viscosum*	10	Ethanol	1:4	40 °C, 8 h	92	[196]
Tallow	*Mucor miehei* (Lipozyme IM60)	10	Ethanol	1:3	45 °C, 5 h	98.3	[196]
Castor oil	*Mucor miehei* (Lipozyme IM)	20	Ethanol	1:3	65 °C, 6 h	98	[196]
Sunflower oil	*Mucor miehei* (Lipozyme)	20	Ethanol	1:11	45 °C, 5 h	82	[196]
Palm kernel	*Pseudomonas cepacia* (PS 30)	10	Ethanol	1:4	40 °C, 8 h	72	[196]
Coconut oil	*Pseudomonas cepacia* (PS 30)	10	Ethanol	1:4	40 °C, 8 h	35	[196]
Jatropha curcas oil	*Pseudomonas cepacia*	10	Ethanol	1:4	50 °C, 8 h	98	[196]
Madhuca indica oil	*Pseudomonas cepacia*	10	Ethanol	1:4	40 °C, 2.5 h	99	[196]
Palm oil	*Pseudomonas fluorescens* (Lipase AK)	20	Ethanol	1:18	40 °C, 24 h	98	[196]
Triolein	*Pseudomonas fluorescens* (Lipase AK)	10	Ethanol	1:3	50 °C, 80 h	70	[196]
Babbasu oil	*Burholderia cepacias* (Lipase PS)	20	Ethanol	1:12	50 °C, 48 h	100	[196]

strength, low solubility in methanol and it can be synthesized from cheap sources like limestone and calcium hydroxide and $CaCO_3$ [167,178,179].

3.5. Enzymatic transesterification

Due to the downstream processing problems caused by chemical tranesterificaiton, enzyme-catalyzed (lipase) transesterificaiton has continued to draw the attention of reseracher's in last several years. Enzyme-catalyzed reactions are less energy intensive, do not promote side reactions and are more environmentally friendly [100]. The realistic problems with enzyme-catalyzed reactions are contamination of the product with residual enzymatic activity and cost of the catalysts. In order to overcome these problems, several potential solutions have been introduced in the last few years, which include the use of immobilized lipases, several types of solvents, acyl acceptors and substrate oil sources. Table 10 details the work carried out for bio-diesel production using various combinations. Much of the research shows great promise, indicating that the use of enzyme catalysts in biodiesel production continues to be a viable option for future. Jegannathan et al. [195] and Al-zuhair [195] studied the effect of two lipases in free and immobilized form, *Rhizomucor miehei* and *Humicola insolens*, in an oil–aqueous biphasic system. The results show that temperature of 40 °C and molar ratio of 6:1 gives the best results. The *Rhizomucor miehei* lipase showed slightly higher conversion, and the immobilized form showed only 30% relative activity after 4 runs. Since lipase in free-form is difficult to reuse and is still relatively expensive compared to alkali catalysts, its use is not very advantageous compared to immobilized lipase and has not been greatly researched in recent times [98,197,198].

3.6. Super critical methanol transesterification

The supercritical methanol transesterification of vegetable oil provides a new way of producing bio-diesel. In the conventional transesterificaiton process high FFAs and water cause negative effects such as soap formation, reduces catalyst efficiency and catalyst consumption, all these effects result in low conversion. To overcome these problems supercritical methanol transesterification is designed to overcome the reaction initiation lag time caused by the extremely low solubility of the alcohol in the triglyceride phase. Supercritical methanol transesterificaiton is the process, in which solvent is subjected to temperature and pressure above its critical point. Under supercritical conditions no longer separate phase exist, only fluid phase is present and at a very high molar ratio of methanol to oil (42:1), the reaction is completed in a short time.

Table 11
Transesterification reaction under supercritical condition.

Oil	Alcohol	Oil to alcohol molar ratio	Temperature and pressure	Reaction time	Ester conversion (%)	Refs.
Sunflower	Methanol	1:40	350 °C, 200 bar	40 min	96	[149]
Jatropha	Methanol	1:40	350 °C, 200 bar	40 min	> 90	[87]
Rapeseed	Methanol	1:42	350 °C, 45 MPa	240 s	95	[200]
Hazelnut	Methanol	1:42	350 °C, NA	300 s	95	[201]
Jatropha	Methanol	1:40	350 °C, 200 bar	40 min	90	[87]
Soybean	Methanol	1:40	350 °C,35 Mpa	25 min	–	[202]
Coconut, palm	Methanol	1:42	350 °C, 19.0 Mpa	400 s	95–96	[203]
Cottonseed	Methanol, ethanol	1:41	350 °C,NA	8 min	98(methanol), 75(ethanol)	[204]
Palm	Methanol	1:45	350 °C,NA	60 min	60.30	[205]
Sunflower	Super critical methanol and ethanol	–	200–400 °C	–	–	[149]
Soybean	Methanol	–	200–300 °C	20 h	–	[108]
Rapeseed	–	1:42	350–400°C,45–65 MPa	10 min	–	[165]
Jatropha	Methanol	–	8.4 MPa, 593 K	4 min	–	[206]
Soybean	Methanol	1:24	12.8 MPa, 280 °C	10 min	98	[207]
Soybean	Methanol	1:24	14.3 MPa, 280 °C	10 min	98	[207]
Soybean	Methanol	1:6	2 MPa, 400 °C	1.6 min	98	[207]
Rapeseed	Methanol	1:24	6 Mpa, 250 °C	10 min	97	[207]
Soybean	Methanol	1:24	10 MPa, 160 °C	10 min	98	[207]
Soybean	Methanol	1:40	13 MPa, 310 °C	12 min	96	[207]
Sunflower	Methanol	1:41	–, 252 °C	6 min	98	[207]
Soybean	Methanol	1:36	24 MPa, 250 °C	10 min	96	[207]
Rapeseed	Methanol	1:24	20 MPa, 280 °C	30 min	95	[207]
Soybean oil	Ethanol	1:40	20 MPa, 350 °C	15 min	80	[197]
Soybean oil	Ethanol	1:40	20 MPa, 350 °C	1.5 cm3 /min	77.5	[197]
Rapeseed oil	Ethanol	1:42	25 MPa, 350 °C	12 min	100	[197]
Rapeseed oil	Ethanol	1:42	40 MPa, 300 °C	20 min	80	[197]
Cottonseed oil	Ethanol	1:41	–, 250 °C	8 min	85	[197]
Linseed oil	Ethanol	1:41	–, 250 °C	8 min	> 85	[197]
Sesame oil	Ethanol	1:40	20 MPa, 325 °C	40 min	100	[197]
Mustard oil	Ethanol	1:40	20 MPa, 325 °C	25 min	100	[197]
Sunflower oil	Ethanol	1:40	20 MPa,400 °C	40 min	100	[197]
Castor oil	Ethanol	1:40	20 MPa, 350 °C	30 min	100	[197]
Linseed oil	Ethanol	1:40	20 MPa, 350 °C	40 min	100	[197]
Palm oil	Ethanol	1:50	20 MPa, 400 °C	30 min	95	[197]
Groundnut oil	Ethanol	1:50	20 MPa, 400 °C	30 min	95	[197]
Pongamia pinata oil	Ethanol	1:50	20 MPa, 400 °C	30 min	90	[197]
Jatropha curcas oil	Ethanol	1:50	20 MPa, 400 °C	30 min	90	[197]

The absence of catalyst makes the glycerol recovery and biodiesel purification much easier, trouble free and environmentally friendly [199]. Due to severe reaction conditions and high operational cost, synthesis of biodiesel by supercritical methanol is not a viable option on the large scale practice in industry. Therefore, for past few years research is focused on to decrease the severity of the reaction condition (Table 11). The use of co-solvents, such as carbon dioxide, hexane, propane or subcritical condition with small amount of catalyst, can decrease the operating conditions. A research group used co-solvents like hexane and carbon dioxide in supercritical methanol method to improve the product yield and 98% yield of methylesters was observed in 20 min at the subcritical condition (160 °C) with 0.1 wt% KOH and methanol to oil ratio of 24:1 [16,22].

3.7. Microwave assisted transesterification

Microwave assisted transesterification is another alternative energy tool which can be used for the production of biodiesel. Due its advantages like reduced reaction time, better yield and environmentally benign compared to conventional heating, it is becoming the obvious choice of many researchers [208,209]. Microwave transfer energy has a tendency to oscillate polar ends of molecules or ions continuously [208,210]. As a result the collision and friction between the moving molecules generate heat. Heat is therefore directly deposited into the reaction media and results in rapid temperature increase throughout the sample [211].

Several studies on microwave assisted transesterification have been reported using different catalyst system and they observed that it offers fast, low oil:methanol ratio, easy operation, reduced energy consumption and minimum by-products (Table 12). However, the major drawbacks of using microwave irradiation for biodiesel synthesis are the scale-up of the process and its safety [49]. Study conducted by Nezihe et al. [212] using microwave yields 93.7% (1.0% (w/w) potassium hydroxide) and 92.2% (1.0% (w/w) sodium hydroxide) transesterified products at 40 °C. Similarly, Michael et al. [222] in their study used continuous-flow microwave assisted transesterification process and found to be more energy-efficient technique than the conventional heating.

3.8. Ultrasound assisted transesterification

The demand for biodiesel in the world is sharply increasing. Thus, increasing the production rate for biodiesel in order to meet the demand seems to be essential. Therefore, new accelerating technologies are of great interest among researchers in this area. Ultrasonic technology has proven to be an effective tool in enhancing the reaction rates in a biphasic (liquid–liquid) heterogeneous reaction system [223]. Ultrasonication provides the mechanical energy for mixing and the required activation energy for initiating the transesterification reaction. Ultrasonication increases the chemical reaction speed and yield of the transesterification of vegetable oils/animal fats. Carmen et al. (2005) in their study reported that use of ultrasonication technique yields highest methyl ester conversion of 95% (1.0% (w/w) NaOH, time 10 min and room temperature) [224,225]. Manh et al. [226] studied the effects of different variables such as molar ratio, catalyst concentration and temperature on ultrasonic transesterification of triolein; the result shows that 20 min ultrasiniaiton time with 1 wt% catalyst concentration, 6:1 M ratio at 25 °C, gives better yield. Similarly, ultrasonic assisted transesterification reactions with other feedstocks under different process condition are shown in Table 13.

4. Technical aspects of biodiesel preparation and quality control

The feedstocks can be used in the production of biodiesel such as vegetable oils, animal fats, or recycled greases contain tri-glycerides, free fatty acids, moisture and other contaminants in various proportions. It is also reported that some crude vegetable oils which contain phospholipids need to be removed in a degumming step. In case of physical refining, the oil is treated at high temperature under vacuum to remove free fatty acids as well as volatile impurities. The process is practiced in the industrial scales where the free fatty acids are separated from the oil to produce producing refined oil with acid value less than 0.2 mg KOH/g. The process is often adopted for palm oil to remove FFA with little loss of oil. Similarly, the feedstocks, which contain considerable amount of free fatty acid (FFA), which is also indicated by the acid value of the oils, are removed in a refining

Table 12
Transesterification reaction under microwave condition.

Oil	Catalyst	Catalyst amount (wt% of oil)	Alcohol	Oil to alcohol molar ratio	Reaction conditions	Ester yield (%)	Ester conversion (%)	Refs.
Cottonseed	KOH	1.5	Methanol	1:6	333 K, 7 min	92.4	–	[208]
Rapeseed	KOH	1	Methanol	1:6	323 K	93.7	–	[212]
Castor	SiO_2/50% H_2SO_4	1	Methanol	1:6	313 K	93.7	–	[213]
	SiO_2/30% H_2SO_4	1	Ethanol	1:6	3 min			
	Al_2O_3/50% KOH	1	Methanol	1:6	30 min			
					25 min			
					05 min			
	NaOH				5 min	–	95	
						–	95	
						–	95	
Triolein	KOH	5	Methanol	1:6	323 K, 1 min	–	98	[214]
	NaOH						98	
Castor	H_2SO_4	5	Methanol	1:12	338 K, 60 min	94	–	[215]
Safflower	NaOH	1	Methanol	1:10	60 °C, 6 min	–	98.4	[120]
algal biomass	KOH	1–3	Methanol	1:9 –1:15	3–9 min	84.15	–	[216]
Pongamia pinnata seed oil	KOH	1	Methanol	1:6	60 °C, 3 min	88.6	–	[217]
	NaOH	0.5				93		
Soybean oil	Nano CaO	3	Methanol	1:7	65 °C,60 min	–	96.6	[218]
Sunflower oil	KOH	–	Methanol	–	100 °C, 9–16 min	10	–	[219]
Algae	KOH	1–3	Methanol	1:9–1:15	60–64 °C, 3, 6, 9 min	50–60	–	[220]
Soybean	KOH	1	Methanol	1:12	70 °C, 65–90 min	–	99	[129]
Ethyl-3-phenylpropanoate	Novozyme 435	0.0024 g/cm^3	n-Butanol	1:2	40–70 °C	–	60–96	[221]

Table 13
Transesterification reaction under ultrasound condition.

Oil	Catalyst	Catalyst wt% of oil	Alcohol	Oil to alcohol molar ratio	Ultrasonic frequency (kHz)	Reaction conditions	Ester yield (%)	Refs.
–	NaOH	0.5	n-Propanol	1:6	28	25 °C, 20 min	92	[224]
					40	25 °C, 20 min	88	
Triolein	NaOH	1	Ethanol	1:6	40	25 °C, < 20 min	–	[226]
Triolein	KOH	1	Methanol	1:6	40	25 °C, 10 min	–	[227]
Soybean	NaOH	1.5	Methanol	–	24	60 °C, 20 min	97	[228]
Fish frying	C_2H_5ONa	0.8	Ethanol	1:6	20	60 °C, 60 min	98.2	[229]
Soybean Oil	NaOH/KOH	0.5	Methanol	1:6	21.5	45 °C, 1 h		[230]
Waste cooking oil	KOH	0.7	Methanol	1:2.5	20	20–25 °C, 15 h	81	[231]
		0.3		1:1.5			99	
Oleic acid	H_2SO_4	5	Ethanol	1:3	40	60 °C, 120 min	95	[232]
FFA from *Oreochromis niloticus*	H_2SO_4	2	Methanol	1:9	40	30 °C, 90 min	98.2	[232]
Rapeseed oil	p-toluenesulfonic acid	3.75	Methanol	1:3.3	20	45 °C, 180 min	11.6	[232]
C_8–C_{10} fatty acid cut	H_2SO_4	1	Methanol	1:10	20	28 °C, 120 min	98	[232]
Soybean oil	KOH	1	Methanol	1:6	20	10 min	99	[232]
Vegetable oils	NaOH	1	–	4:4	20	–	–	[232]
Soybean oil, castor oil, peanut oil, soybean oil	NaOH	1	Methanol	1:5	20	60 °C, 1.5 min	96	[232]
Rapeseed oil	NaOH	2	Methanol	1:7	24	60 °C, 20 min	96	[232]
Rapeseed oil	NaOH	0.5	Methanol	1:3.3	–	45 °C, 20 min	85	
Soybean oil	NaOH	1	Methanol	1:6	20	30 °C, 10 min	90.2	[232]
Beef tallow	KOH	0.5	Methanol	1:6	20	60 °C, 1.17 min	92	[232]
Waste vegetable oil	KOH	1	Methanol	1:6	20	65 °C, 5 min	98.94	[232]
Waste cooking oil	KOH	0.5–1.5	Methanol	1:6	20	30 °C, 30 min	96.78	[232]
Triolein	KOH	1	Various alcohols	1:6	40	25 °C, 15 min	92	[232]
Jatropha curcus oil	$NaSiO_2$	3	Methanol	1:9	24	Ambient temperature, 15 min	99	[232]
Rapeseed oil	Mg/MCM-41/10 Mg-Al Hydrotalcite/10 10 K/ ZrO2/10 20 K/ZrO2/20	10, 20	Methanol	1:7	24	60 °C, 300 min	89, 96, 70, 83	[232]
Rapeseed oil	KF/CaO	3	Methanol	1:12	20	65 °C, 60 min	98.7	[232]
Palm oil	CaO	3	Methanol	1:12	20	65 °C, 60 min	77.3	[232]
	SrO	3	Methanol	1:9	20	65 °C, 60 min	95.2	
	BaO	3	Methanol	1:9	20	65 °C, 60 min	95.2	
Tung oil	KOH	1	Methanol	1:6	25	20–30 °C, 1–30 min	87–91	[233]
Sunflower oil	NaOH	0.5–2	Methanol	1:3–7,10,15	28	Ambient temperature, 50 min	80–95	[234]
Recycled oil	NaOH	0.6, 1	Methanol	1:6, 1:12	35	6o °C, 60, 75 min	89	[235]
Jatropha oil	KOH	5	Methanol	1:4	24	50–180 °C, 30 min	84.5	[236]
Soybean oil	NaOH	1	Methanol	1:6	20	60 °C, 1, 2, 5, 10, 1 min	97.7	[237]
Jatropha oil	NaOH	3	Methanol	1:9	24	Atmospheric, 15 min	98.53	[238]
Canola oil	KOH	0.7	Methanol	1:5	20	70 °C, 50 min	99	[239]
Soybean oil	KOH	0.5	Ethanol	1:12	20	Ambient temperature, 60 min	95.6	[240]

step and excess free fatty acids can be removed as soaps later in a pretreatment step. In order to estimate the FFA percentage in the oil or fats, titration is performed and if the percentage of FFA is above 2.5 wt%, pretreatment is used to reduce the FFA content. This step also determines the amount of caustic soda required in the neutralization step. In addition to that deodorization is another important step in the treatment of raw materials. During this process raw material is treated with steam in order to eliminate FFAs, aldehydes, unsaturated hydrocarbons and ketones, all of which cause undesirable odors and flavors in the oil [73].

Esterification of FFA with methanol or glycerol is conducted for the pretreatment of high FFA oils. The esterification of FFA with methanol is catalyzed by an acid i.e. sulfuric acid to produce methyl esters. There are high chances that the catalyst used during the process may contribute to the sulfur content of biodiesel. This process is rarely practiced in the industries. Another method of pretreatment

is to treat with glycerol and heat it up to 200 °C. The glycerol will react with the FFA to form monoglycerides and diglycerides. The esterification of FFA with glycerol gives rise to glyceride and water. The reaction is conducted at high temperature under vacuum so that water eliminated is removed from the reaction vessel shifting the equilibrium to favor the esterification. The advantage of this approach is that no alcohol is needed during the pretreatment. There is no loss of oil in this process of pretreatment. However, the drawbacks of this method are its high temperature requirement and relatively slow reaction rate [86]. Neutralization is performed where the feedstocks contain considerably amount of FFA. The oil is treated with appropriate amount of alkali such as NaOH solution, the amount of sodium hydroxide is calculated from the acid value or FFA of the oil. During the process FFA are converted to soap which is removed by water washing and the loss of oil during neutralization is high.

After pretreatment, transesterification of the pretreated oil is carried out by the use of alkali catalysts such as methoxides and hydroxides of sodium and potassium. Recently, there is an increased interest in new technologies related to mass transfer enhancement as mentioned above. Glycerol is separated by gravity separation or by the use of centrifuge. The crude biodiesel after the reaction is post processed to remove the residual catalyst, methanol, water and other impurities to produce final biodiesel.

4.1. Quality control

The quality of biodiesel depends on various factors and usually ASTM standards are followed all over the world to check the quality of biodiesel. The properties such as oxidation stability, cloud point, iodine value, cold soak filtration, linoleic acid methyl ester content, poly-unsaturated fatty acid methyl ester content of biodiesel are dependent upon the quality of the feedstock. The density, viscosity, acid value, distillation property are dependent on feedstock as well as the reaction conditions or the extent of reaction. Among the general parameters for biodiesel, the viscosity controls the characteristics of the injection from the diesel injector. The viscosity of fatty acid methyl ester can go very high levels and hence it is important to control it within the acceptable level to avoid negative impacts on fuel injector system performance. Therefore, the viscosity specifications proposed are nearly same as that of the diesel fuel. The methyl ester content, monoglyceride, diglyceride, triglyceride, total glycerol content depend on the extent of completion of the reaction. Phosphorus and sulfur contents are dependent on vegetable oil quality as well as extent of completion of degumming and pretreatment process, respectively. The sulfur content may be contributed by the sulfuric acid catalyst during esterification with methanol. Rest of the properties mostly depends on the post processing of biodiesel after transesterification.

5. Conclusion

Bio-diesel is a renewable fuel produced mainly from plant/vegetable oils and waste lipids. Due to the apprehension on the availability of recoverable fossil fuel reserves and the environmental problems caused by the use of those fossil fuels, considerable attention has been given to bio-diesel production as an alternative to petro diesel. Most of the biodiesel that is currently made uses edible oils. In Indian context, the use of edible oils for biodiesel is economically not viable since we depend on other countries to fulfill the edible oil demand. Hence, focus needs to be shifted to non-edible oilseed plants available in India. The non-food lipid feedstock has the potential for the production of biodiesel that can partially supplement to the growing demand for diesel. The article describes the feasibility of biodiesel from non-edible oil resources which are available in India including microalgae, the technical and commercial aspects.

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